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6. AUTHOR(S)
Miriam Deutsch

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Department of Physics
University of Oregon

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13. ABSTRACT (Maximum 200 words)

Our Research focuses on the synthesis and characterization of new optical materials. Materials periodically patterned on optical wavelength scales, such as three-dimensional photonic crystals hold great promise for generating and controlling optical signals. Examples include enhanced optical sensing capabilities of nano-structured materials, and nonlinear photonic crystals for optical logic applications.

Of particular interest to us is a new class of photonic materials, known as metallo-dielectric photonic structures. These materials are of immense interest due to the unusual dispersive effects present in metals, as well as greatly enhanced optical nonlinearities which have been demonstrated in these systems. These unique phenomena are mediated by the excitation of surface plasmon polaritons at the metal-dielectric interfaces. Such low-dimensional excitations are confined to dimensions much smaller than the wavelength of light, thus facilitating optical interactions at sub-diffraction-limit volumes. The rich optical phenomena offered by metallo-dielectrics therefore holds tremendous potential for novel optical materials, with unique spectral response and enhanced optical nonlinearities.

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I.a) Percolative Metal-Dielectric Nanoshells – Single Particle Optics

We have been focusing on fabricating metallodielectric core/shell particles with controlled surface morphologies (either smooth or highly disordered nanoshells). We have been using mainly silver as the metal for the nanoshell, although we have also fabricated some gold shells. These materials are currently being tested for optical sensing applications. We are addressing their linear light-scattering properties, as well as their nonlinear optical response. In addition, we have fabricated ordered 3D metallodielectric photonic crystals using nano-silver coated colloidal silica spheres, and are currently characterizing their optical properties.

We have modified conventional electroless deposition techniques to fabricate nanocrystalline, disordered silver nanoshells on colloidal silica cores [1]. Silica spheres with highly controllable size distributions and diameters in the range of 100nm-1000nm are grown using well-established sol-gel synthesis methods [2]. Using a variation of the Tollen's reaction, granular silver nanoshells deposit

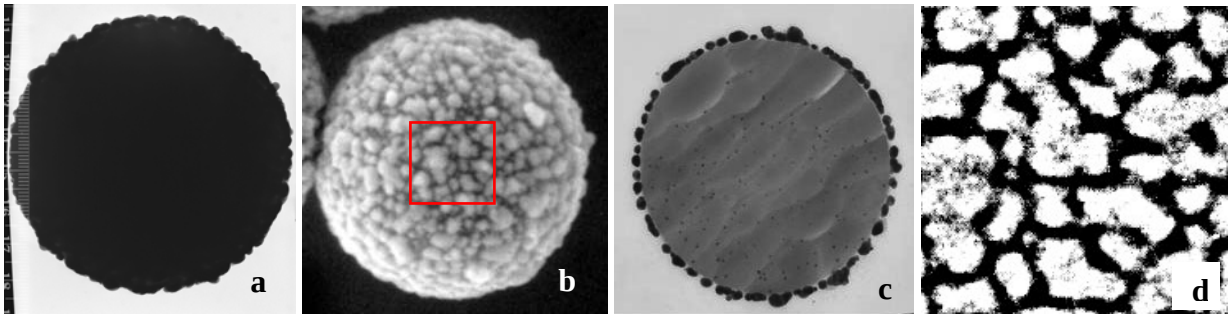

Figure 1. (a) Transmission electron (TEM) and (b) scanning electron (SEM) micrographs of silica spheres 1μm in diameter, coated with a 20nm fragmented silver shell using the modified Tollen's procedure described above. (c) Cross-sectional TEM of a single coated sphere, showing a uniform granular coating. (d) Magnified view of the region shown inside the red square in (b). It is seen that most metal clusters are not in contact, and therefore this shell is below percolation.

uniformly only at the surfaces of the spheres. Fig. 1 shows typical results for silver-coated silica spheres fabricated in our lab. The metal coating is extremely granular, yet it is also highly uniform in thickness such that the spherical shape and polydispersity of the colloids are conserved to a very high degree. To confirm this we have measured the size of ~250 coated spheres using standard transmission electron

microscopy (TEM) sizing techniques. As an example, using silica spheres 1μm in diameter we measured a diameter of 1.05μm after silvering, with a polydispersity of 4%. Such rough metallic nanoshells are particularly suitable for surface-enhanced SHG and SERS studies. In contrast to other core-shell fabrication techniques, we do not utilize any surface functionalization of the spheres to enhance the adhesion of silver to the silica. This is so since the presence of organic surface-functional groups may be detrimental when NLO properties of the particles are probed under intense laser illumination. Nevertheless, we find that the silver shell binds strongly with the untreated silica surfaces. The spheres in Fig. 1 have undergone several cycles of centrifugation and ultrasonic re-dispersion, and still retained the metal coating. Thus this system constitutes a pure two component, inorganic metallodielectric structure, suitable for optical applications.

An interesting feature of these nanoshells is their discontinuous, granular composition. High resolution SEM studies show that shells of ~20nm thickness have a metal island filling fraction, p of 0.5 – 0.6, which is below the known 2D metal-insulator phase transition critical value of p_c= 0.68 [3]. The metal island size is typically 5 – 10nm. We have also measured the fractal dimension of the shells (by digitizing filtered SEM images and using a box-counting algorithm,) and found it to be in the range 1.7-1.8. This is very close to the measured value of ~1.78 for 2D percolative metal films [4].

To determine whether these shells are percolative we measure their DC conductivity. Our current experimental setup does not allow testing single spheres. We therefore measure the resistance of dense sphere aggregates instead. We do this by drying several drops of the concentrated sphere suspension in a narrow gap (~100μm) separating two gold electrodes evaporated on a glass microscope slide, and measure the resistance across the gap. We observe that shells of the thickness shown in Fig. 1 (~20-30nm) exhibit very high resistance ($>1\,\mathrm{M}\Omega$,) while much thicker shells (~100nm) show metallic contact resistance ($<2\,\Omega$.) These findings suggest that the thin metal shells we are fabricating are below percolation, and consequently could exhibit the unique dielectric response typical to such metal-dielectric composites near the percolation threshold.

We recently verified this hypothesis from light scattering experiments [5]. Extinction spectra taken of dilute coated-sphere suspensions reveal oscillatory behavior as shown in Fig. 2(a). We model this response using Mie scattering theory modified for core/shell spherical scatterers [6,7]. It is important to note that this formalism requires knowledge of the dielectric function of both core and shell materials, while in our case the latter, ε_m, is not known. The usual approach for modeling the extinction coefficient

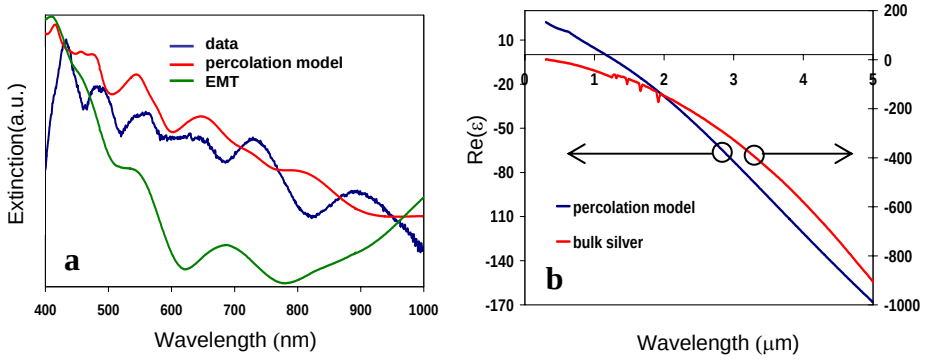

Figure 2. (a) Comparing extinction spectra calculated using a percolative model and EMT. (b) Dielectric function of percolative thin shell as calculated from our model, compared to that of bulk silver.

using tabulated values for bulk silver [8] exhibited serious discrepancies and failed to reproduce the experimental data. We therefore construct ε_m from first principles, similar to methods used to model thin disordered metal films near the percolation threshold [9], and use the resulting function to calculate the extinction coefficient as shown here.

The agreement is seen to be very good. For comparison, we also plot in Fig. 2(a) the extinction coefficient obtained using classical Bruggeman effective medium theory (EMT) for the shell. This model takes into account geometrical averaging based only on nanoparticle shape and volume fraction in the shell, but neglects frequency-dependent (AC) coupling between silver grains at optical frequencies [10]. It is clear that while EMT qualitatively agrees with experiment, the percolation-based model provides a much closer description of this metallodielectric system.

Our findings showing the percolative nature of silver nanoshells have important consequences. Fig. 2(b) shows the effective dielectric function ε_m obtained from the percolation model, as well as the known dielectric function for bulk metallic silver. The main difference between the two is that in the visible and mid-IR the percolative model yields *positive* values for ε_m, that is *dielectric behavior*. The function is seen to cross over to negative values only at $\lambda \sim 1.2$ μm. We note that the overall trend of ε_m is identical to that of the bulk silver function. This model is consistent with our observations: the sharp resonances in Fig. 2(a) are a clear example of Mie resonances in a composite core/shell dielectric (non metallic) system. Moreover, the broad plasmon resonance observed in metal nanoshells [8] is absent, indicating *no metallic behavior at these frequencies*. This is in striking difference to plasmon resonances observed in uniform metal nanoshells.

We find that although Fig. 2(a) shows qualitative agreement between EMT and the percolation model in the visible range, major discrepancies are expected in the IR. While dielectric behavior is observed in the visible, the crossover to metallic behavior predicted by our model should manifest in a tunable plasmonic response in the IR [5]. We are currently conducting additional spectroscopic

experiments aimed to address this predicted IR absorption. These nanoshells are unique, since they are predicted to exhibit an insulating dielectric response in the visible and a metallic response in the IR.

Other silver nanoshell fabrication techniques [8,11] were also reproduced in our lab. The advantage of these methods is that they allow the fabrication of thin, complete metallic nanoshells as in Fig. 3. It is advantageous to have fabrication capabilities not only of nanoshells of controlled thickness, but also of controlled roughness as we developed here. It is known that the linear SPR, as well as surface-enhanced NLO phenomena depend strongly on the morphology of the metallic surfaces. Other metals such as gold [12,13], as well as metal oxides [14] may be deposited using similar methods to form nanoshells of varying morphologies. We expect that by tuning the shell morphology enhanced sensing capabilities will emerge. Ultimately, we are aiming at understanding the yet unexplored sensitivity limits of a single core/shell system.

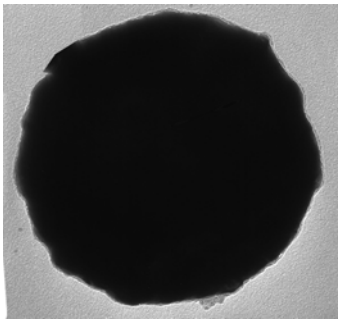

Figure 3. TEM of silica sphere 250nm in diameter, with a complete silver nanoshell [8].

I.b) Nonlinear Optical Response: Surface Enhanced SHG

We have recently observed strong second-harmonic and supercontinuum signals generated from disordered aggregates of 1μm silver-coated spheres [1]. When illuminated with 120fs laser pulses at 860nm, frequency-doubled signals at 430nm were detected, in addition to a very broad and intense supercontinuum spanning the entire visible and NIR spectrum, as shown in Fig. 3. Since silver does not generate second harmonics in the bulk due to its centrosymmetric crystalline structure, frequency doubling originates only at the nanocrystals' surfaces and is therefore a surface-enhanced effect [15,16]. Comparing to measurements using uncoated silica spheres, we have ascertained that the strong SHG is due to the presence of silver. Individual silvered spheres probed under a high-resolution optical microscope also exhibit the same nonlinear response.

Recent experiments in hyper-Rayleigh scattering from silver nanoparticles suggest that their hyperpolarizablities greatly exceed those of highly NLO organic chromophores [17]. Compared to a standard such as $LiNbO_3$, these materials may therefore possess nonlinear coefficients which are orders of magnitude larger. It is hence essential that we understand the evolution of the coherent NLO response in nanocrystalline silver shells. We are currently conducting detailed studies of the nature of these signals.

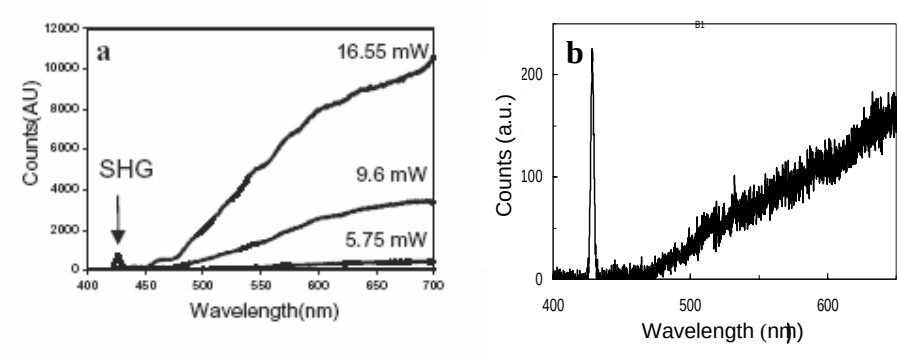

Figure 3. (a) SHG and super-continuum generated from aggregate of coated spheres at different pump powers. (b) SHG and continuum signals from a single silver-coated sphere.

We aim to understand conversion efficiencies in dispersed systems as well as self assembled metallodielectric photonic crystals. We are addressing the dependence of SHG on morphology and core diameter, and ordering of the spheres. Our observations so far suggest that the SHG from metal nanoshells makes them excellent candidates for optical sensing applications.

I.c) Mesoscale Plasmonics – Theory and Modeling

As the need for integration of compact lightwave devices is growing, it is necessary that we develop an understanding of the fundamental properties of SPP propagation and manipulation in these environments. We have been studying the performance of several elementary plasmonic components from which complex microplasmonic circuits may be constructed. We have been conducting theoretical studies and numerical simulations of planar metallodielectric plasmonic structures. Interestingly, while significant progress has been made in understanding surface plasmon polariton (SPP) scattering and propagation in nano-structures [18-20], certain fundamental issues pertaining to their guiding on mesoscopic metallic films and waveguides remain unknown. In particular, quantifying guiding and energy losses in SPPs propagating around bends in metal-dielectric interfaces (such as in 3D MDPCs) is of great importance, as it should set a limit on feature size in plasmonic-circuit devices such as SPP-based integrated nano-sensors.

We have previously analyzed SPP propagation efficiencies about a rounded edge as shown in Fig. 4(a) [21]. We have shown analytically that in the short-wavelength limit, even in the presence of bend-induced radiation losses, transmittance of SPPs around a finite-radius metallic bend may exhibit lower losses than on flat surfaces. In order to increase the efficiency of this structure we consider the geometry shown in Fig. 4(b). A cylindrical hole of radius *R* is placed in close proximity to two flat metal interfaces joined by an abrupt 90° edge. Positioning the resonator within the skin depth of the metal allows efficient

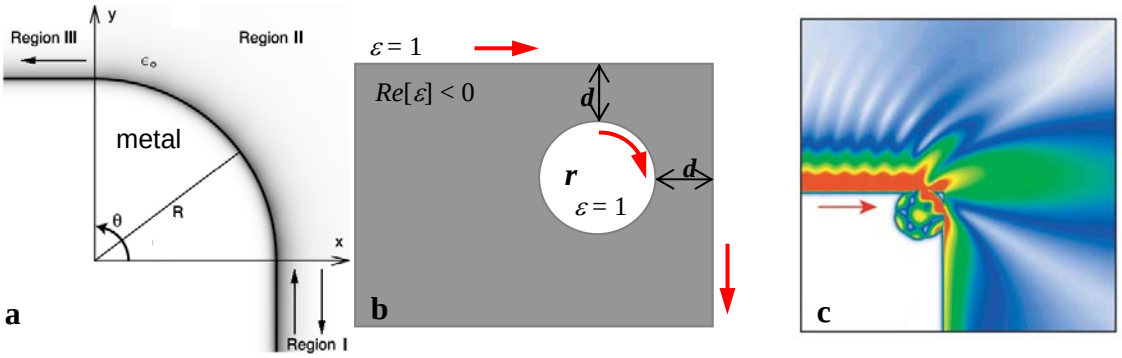

Figure 4. (a) Top view of the bend geometry. SPPs are incident from Region I onto a corner of radius R and bend angle θ. Output consists of back-reflection into Region I and transmission into Region III. (b) Schematic of SPP coupler embedded near a sharp edge. SPPs are incident from the left along the red arrow, couple to the resonator and couple out (c) Numerical finite-difference time-domain (FDTD) simulation showing magnitude of the magnetic field for d = 8.4 *nm*, λ = 600*nm* and $2r$ = 756*nm*. A significant portion of the field couples into the resonator. Complications arising from the excitation and interference of multiple resonator modes are avoided by keeping the radius small $(2r < 1.5\lambda)$, so a maximum of two resonator modes may be excited.

excitation of the resonator modes by the incident SPPs, and subsequent outcoupling. The dielectric void acts a directional coupler, and the evanescent wave coupling scheme is analogous to optical coupling between microring resonators and dielectric waveguides [22]. Fig 4(c) shows results of numerical simulations for a typical resonator coupled system. In general, we find that the efficiency of propagation around sharp bends increases from 7% to 21% when a resonator is properly incorporated into the metal in the vicinity of the bend [23]. As with all ring resonators, mode coupling is highly sensitive to the values of both d and r. These results shed new light on the mesoscopic behavior of SPPs, and should play an important role in the design and optimization of SPP devices. In our present study we aim to develop formalism for efficient control and manipulation of SPPs at structured interfaces. The goal is to devise a set of highly sensitive plasmonic elements which can be utilized to construct compact integrated sensors.

A model system which we have been studying recently is the mesoscopic metal-dielectric-metal (MDM) sphere. This is described by a spherically symmetric Bragg resonator [24] comprising thin, alternating layers of dielectric and metal shells around a spherical metal core. We have solved Maxwell's equations in spherical coordinates and obtained the dispersion of such MDM spheres. Using a Mie scattering multipole expansion for plane-wave scattering in the visible/NIR we have also obtained the absorption cross-sections [25], as shown in Fig. 5 (a). The spherical nanoshell is known to exhibit a

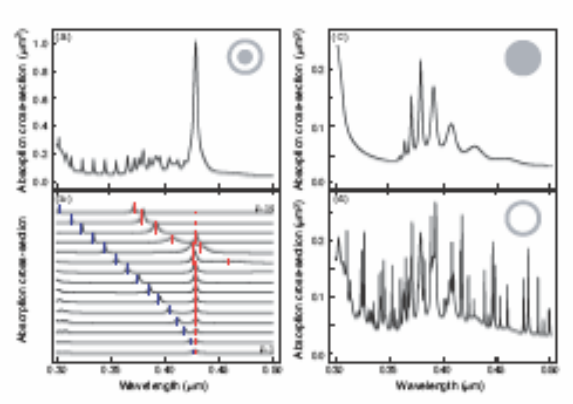

FIG. 5. (a) Optimized absorption cross-section spectrum of MDM sphere with a total radius of 500nm. (b) Mode decomposition of the spectrum from angular momentum number l = 1 (bottom) to l = 15 (top), showing contributions from transverse electric modes (blue hatches), and transverse magnetic modes (red hatches). The flat dispersion band is clearly visible. (c) Absorption spectrum of a metal sphere of radius equal to that in (a). (d) Absorption spectrum of a dielectric sphere with metal shell equal in thickness to that in (a). All insets show schematic cross-sections of the particles [25].

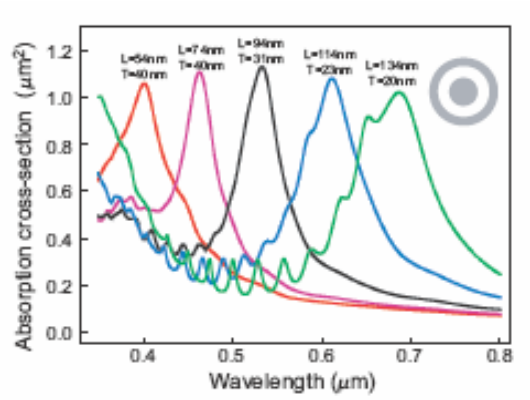

FIG. 6. Absorption spectra of silver-titania MDM sphere with radius of 500nm and various dielectric shell thickness L. The value of the metal shell thickness, T is chosen to maximize the peak height. With proper choice of shell thicknesses the absorption spectrum is tunable over the entire visible spectral range. The width of the absorption line remains smaller by several fold from that of the dipole resonance of nanoscale core/shell particles [25].

strong plasmon resonance which may be tuned through appropriate design of the core/shell ratio of the particle, as shown in Fig. 6. We find that this tunable plasmon resonance is further enhanced by at least one order of magnitude in our MDM structures. These structures may be understood as the rotational analogs of layered planar metal-dielectric-metal films [26], with angular momentum replacing linear momentum. For large enough particles (0.5-1μm) the number of angular momentum eigenmodes excited by plane-wave scattering is large enough to give rise to plasmonic mini-bands in the visible, as shown in Fig. 5(b). With proper design of the nanoshell thickness ratios, we find that the dispersion may be rendered nearly flat over a wide range of angular momentum values, resulting in a giant and narrow plasmon resonance. An interesting attribute of the nearly flat band is its slope, which we find is always negative over a large range of angular momenta, indicating left-handed optical response in this new meta-material [27].

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Participating Scientific Personnel:

Graduate Students: Keisuke Hasegawa, Charles A. Rohde.

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- *Plasmon-Assisted-Transparency in Multi-Shell Metallodielectric Resonators*, C. A. Rohde, K. Hasegawa, M. Deutsch.

Submitted:

- *Silver Nanoshells – Fabrication and Nonlinear Optical Response*, J. P. H. Bouwman, S. M. Emmons, C. A. Rohde, A. Chen, M. Deutsch, under revision.

- *Enhanced Surface Plasmon Resonance Absorption in Metal-Dielectric-Metal Layered Microspheres*, K. Hasegawa, C. A. Rohde, M. Deutsch, arXiv: physics/0511246 (2005); submitted for publication in Opt. Lett. (2005).

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- *Surface Plasmon Polariton Propagation around Bends at a Metal-Dielectric Interface*, K. Hasegawa, J. U. Nöckel, M. Deutsch, *Appl. Phys. Lett.* **84**, 1835 (2004).

In conference proceedings:

- *Surface Plasmon Polariton Propagation around Bends at a Metal-Dielectric Interface*, K. Hasegawa, J. U. Nöckel, M. Deutsch, *Proc. SPIE* **5728**, 184 (2005).

- *Percolation-Enhanced Supercontinuum and Second-Harmonic Generation from Metal Nanoshells*, C. A. Rohde, K. Hasegawa, A. Chen, M. Deutsch, Organic and Nanocomposite Optical Materials. Symposium (Materials Research Society Proceedings Vol. 846), Warrendale, PA (2004) (refereed).

Papers presented at meetings but not published in conference proceedings:

- C. Rohde, K. Hasegawa, M. Deutsch, *Coherent light scattering in percolative metallodielectric nanoshells*, Conference on Lasers and Electro-Optics (CLEO/QELS), Baltimore, May 2005.

- C. Rohde, A. Chen, K. Hasegawa and M. Deutsch, *Percolative metal nanoshells Percolative metal nanoshells for metallodielectric photonic crystals*, Integrated Photonics Research and Applications and Nanotechnology for Information Systems Topical Meetings, San Diego, April 2005.

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- J. Bouwman, C. Rohde, M. Deutsch, *Self-assembled metallo-dielectric photonic crystals with composite silver/silica colloidal spheres*, Conference on Lasers and Electro-Optics (CLEO), Baltimore, June 2003.